IS 4285 (1967): Method for volumetric determination of calcium [CHD 1: Inorganic Chemicals]

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# *Indian Standard*

# METHOD FOR VOLUMETRIC DETERMINATION OF CALCIUM

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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# *Indian Standard*

# METHOD FOR VOLUMETRIC DETERMINATION OF CALCIUM

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## 0 FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 12 September 1967, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

**0.2** The precipitation of calcium as oxalate and its determination by dissolving the washed precipitate in hot dilute sulphuric acid and titrating with standard potassium permanganate is widely used for the determination of calcium. The accuracy of the volumetric method compares favourably with that of the gravimetric method. The volumetric method is more rapid when many samples are to be analysed.

**0.3** This standard intends to achieve uniformity of the method for the volumetric determination of calcium when prescribed in Indian Standards for analysis of chemical products.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value(s), observed or calculated, expressing the result(s) of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value(s) should be the same as that of the specified value(s) in this standard.

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## 1. SCOPE

**1.1** This standard prescribes the method for the volumetric determination of calcium by the potassium permanganate method.

## 2. QUALITY OF REAGENTS

**2.1** Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Rules for rounding off numerical values (*revised*).
†Specification for water, distilled quality (*revised*).

## 3. REAGENTS

**3.1 Ammonium Oxalate Solutions** — (a) saturated, and (b) 1 percent (*w/v*).

**3.2 Methyl Red Indicator Solution** — Dissolve 0·15 g of methyl red in water and dilute to 500 ml.

**3.3 Dilute Ammonium Hydroxide** — approximately 5 N.

**3.4 Dilute Sulphuric Acid** — 1 : 4 (*v/v*).

**3.5 Standard Potassium Permanganate Solution** — 0·02 N.

**3.6 Concentrated Hydrocholoric Acid** — conforming to IS : 265-1962*.

**3.7 Dilute Hydrochloric Acids** — (a) 1 : 4 ( *v/v* ), and (b) 1 : 100 ( *v/v* ).

## 4. PROCEDURE

**4.1** Take a clear weakly acidic solution as prepared in the relevant Indian Standard containing not more than 50 mg of calcium. Dilute to 200 ml and add 2 to 3 drops of methyl red indicator. Add 5 ml of concentrated hydrochloric acid. Heat to boiling and add with constant stirring an excess of hot ammonium oxalate solution. Heat to 70° to 80°C and add with constant stirring dilute ammonium hydroxide ( 5 N ) dropwise until the colour changes from red to yellow. Allow the solution to stand at 70° to 80°C for one hour and filter through filter paper. Wash the precipitate with cold ammonium oxalate solution ( 1 percent ). Dissolve the precipitate in 50 ml of hot 1 : 4 hydrochloric acid. Wash the paper with hot 1 : 100 hydrochloric acid and dilute to 200 ml. Carry out precipitation of calcium oxalate as described above. Allow the solution to stand for 4 hours and then filter through a filter paper. Wash the precipitate with cold ammonium oxalate solution ( 1 percent ) until it is free from chlorides and then with minimum quantity of hot water until free from oxalates. Pierce the apex of the filter paper with a stirring rod and wash down the bulk of the precipitate into a conical flask. Dissolve the precipitate by pouring warm dilute sulphuric acid. Finally, wash the filter paper thoroughly with hot water. Add about 30 ml of dilute sulphuric acid to the washings, dilute to about 200 ml, heat to 60°C and titrate while hot with standard potassium permanganate solution to an end point of pink colour persisting for 30 seconds.

NOTE — In the preparation of the solution it shall be observed that metals upto Group III B are removed and where magnesium is known to be absent a single precipitation shall suffice.

*Specification for hydrochloric acid ( *revised* )

## 5. CALCULATION

**5.1** Calcium ( as Ca ), percent by weight $= \frac{A \times N \times 2{\cdot}004}{W}$

where

$A$ = volume in ml of standard potassium permanganate solution required for titration,

$N$ = normality of standard potassium permanganate solution, and

$W$ = weight in g of material contained in the test solution.

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